

NPL Polarized Source Group Technical Note # 90-2

How to Charge the NF₃ Leak Valve on the Illinois/CEBAF Polarized Electron Source

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This note details the procedure followed to charge the NF₃ leak valve in the original version of the Illinois/CEBAF polarized electron source in which the NF₃ was "stored" in a short length of "pinched- off" copper tube. It is based on the procedure originally developed at SLAC for the Peggy II source. See Technical Note # 90-2A for a revised version of the procedure appropriate for the reusable NF₃ storage system now in use.

Materials

- 1. Two 12" lengths of cleaned 1/4" OD copper refrigerator tubing (see note 1).
- 2. Four cleaned 1/4" SS Swagelock fittings and ferrules (see note 1).

Procedure

- 1. Check that the right angle valve (see point 1 in the attached figure) above the NF₃ sorption pump (2) is closed and cool the sorption pump down for about one hour. (Numbers here and in the following steps refer to points identified in the figure attached.)
- 2. Take an RGA spectrum to document the quality of the vacuum if one is not already available from earlier work on the source.
- 3. Connect a clean, 12" long piece of 1/4" OD copper refrigeration tubing between one side of the Balzers leak valve (3) and the 4-way cross attached to the sorption pump valve (1) (see note 6).
- 4. Connect another 12" length of clean 1/4" OD copper tubing between the other side of the leak valve and the tee (4) which connects to the NF₃ tank (5) and its regulator to the nitrogen heater and its valve (6).
- 5. Close the valve isolating the NF₃ tank and open the up-to-air valve (7) on the 4-way cross by the sorption pump. Flow nitrogen through the system and turn on the nitrogen heater. Place a thermocouple (8) on the copper tube between the sorption pump the leak valve, and heat to 140 C; you will have to wrap the tubing with fiberfrax.
- 6. Bake out the tubing for 2-3 hours using the flowing hot nitrogen as the heat source.

- 7. Turn off the nitrogen heater, and continue to flow nitrogen through the system until it cools to room temperature.
- 8. Close the Nupro valves on the nitrogen heater and the up-to-air valve, and begin to evacuate the system by opening the valve (1) to the sorption pump. Monitor the pressure with the convectron gauge. If the pressure does not fall to a few millitorr quickly, one of the Swagelock fittings is probably leaking. If a leak is observed, tighten each Swagelock fitting gently until the pressure falls to a few millitor. Then, as a check, close the valve to the sorption pump and verify that the pressure does not rise quickly. If it does, you still have a leak; try tightening the Swagelocks a bit more and repeating this step. If not, you are ok and can move on to the next step.
- 9. Backfill system with 2-3 psi of NF₃.
- 10. Repeat evacuation and backfill (steps 7 and 8) two or three times.
- 11. Charge system to 2-3 psi of NF₃.
- 12. Use Scotch Brite to clean the regions on the two copper tubes connected to the Balzer's leak valve about 4" from the valve (ie. where the pinch-offs will be made) to remove any oxides that have formed. Then pinch off the copper tubing on both sides of the leak valve, and cover the sharp edges with RTV sealant (see note 5).
- 13. After sorption pump has reached room temperature, purge it by running nitrogen through the up-to-air valve at a pressure of 7 to 8 psi. After the pressure relief valve pops, let the nitrogen run for an hour to flush out the NF₃ prior to baking the pump for future use.
- 14. Check that the NF₃ leak valve tubes have been properly charged and sealed by taking an RGA spectrum in the gun with the leak valve opened to the position for normal operation during cathode fabrication. Open the leak valve until the pressure in the gun rises to the point where the main ion pump current has stabilized at 3.5 μA. Then take a second RGA spectrum. Typical spectra showing what you should expect to see before and after the leak valve is opened are attached.

Notes

- 1. The copper tubing and the swagelock fittings should be cleaned following the standard procedure of fifteen minutes each of ultrasonic cleaning in successive baths of 1,1,1 trichloroethane, then acetone, then methanol, and then blow-dryed with dry nitrogen. If you have a supply of copper tubing that is known to be clean and dust-free, it does not need to be cleaned if it has been stored with the ends capped.
- 2. NF₃ is noncorrosive to common metals below 70 C. Provide adequate ventilation when handling inhalation of large quantities of gas can cause suffocation.
- 3. Always be sure to purge the NF₃ from the leak valve prior to a system bakeout.
- 4. The small sorption pump used for charging the NF₃ system must be reserved for this purpose only.
- 5. Pinch off the copper tubing using a model POD-375 pinchoff tool, available from CHA Industries.
- 6. To tighten the Swagelock fittings, first tighten them by hand (ie, "finger-tight." Then turn them 1 1/4 turns past this point. Do NOT overtighten these fittings as you may damage the fittings on the NF₃ leak valve.
- 7. NF₃ is purchased from Air Products Specialty Gas, their item # J05-Y-40032. The regulator is their part # E11-TC334A.





